

# Synthesis, Characterization and Biological Activities of Tellurium(IV) Complexes of Bidentate Schiff Base Derived from 5-Chlorosalicylaldehyde and 3-Aminopyridine

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## ABSTRACT

A series of tellurium(IV) complexes were synthesized from Schiff base i.e. 5-chlorosalicylaldehyde-3-aminopyridine Schiff base (3-APY-{5-ClSalH}). Obtained Schiff Base and their tellurium(IV) complexes have been characterized by elemental analyses, conductivity measurements, FT-IR and <sup>1</sup>HNMR spectral studies, suggests that Schiff base ligand behave as monobasic ON bidentate at tellurium metal centre. Also, the Schiff base and some of their tellurium(IV) complexes were tested for antifungal and antibacterial activities using Broth Dilution Method.

**Keywords :** 5-Chlorosalicylaldehyde, Monobasic, Schiff Base, Tellurium(IV) Complexes, Biological Activity.

## I. INTRODUCTION

Schiff bases derived from aldehyde or ketone with imines[1]. It is known that the existence of metal ions bonded to biologically active compounds i.e substituted salicylaldehyde possesses higher activities[2-5]. Chelating ligands containing O, N and S donor atoms show broad biological activity [6] particularly when the group such as -OH / -SH are near to azomethine group, resulting in the formation of five or six membered ring complexes[7-12]. Schiff bases are reported to have biological activities like antibacterial [13-21], antifungal [13,15-18,22], antitumor [16,23,24], antiviral [25-27], anti-HIV [28], herbicidal[29] and anti influenza A virus[30] activities. Also, Tellurium(IV) chloride and organytellurium(IV) Chlorides known to form adducts with amides[31,32] and thiourea[33], thus reflecting its acceptor behaviour[7,8,34-59]. In view of this, we have investigated of Schiff base derived from 5-Chlorosalicylaldehyde and 3-aminopyridine towards organytellurium(IV) chlorides which are known to act as lewis acids. These complexes have been

examined for their antimicrobial activity against different strains of bacteria and fungii.

## II. METHODS AND MATERIAL

All chemicals used were of Analytical Reagent grade. All preparations were carried out under an atmosphere of dry N<sub>2</sub> atmosphere. The solvents were purified by standard method[60,61] before use. The purity of compounds was checked by TLC using Silica gel-G (Merck). Melting points were determined in open capillary tube and are uncorrected.

Carbon, hydrogen and nitrogen analyses were obtained microanalytically from SAIF, Panjab University Chandigarh on a ThermoFinnigan CHNS analyser. Conductivity was measured in DMSO at 25±2°C with a dip type conductivity cell on a microprocessor based conductivity bridge type MICROSIL.

Infrared spectra (4000-40 cm<sup>-1</sup>) were recorded in KBr and Polyethylene pellets for Mid-IR and Far-IR respectively, on a F.T. Infra-Red Spectrometer Model

Nicolet IS50 (Thermo Scientific). Proton NMR Spectra were recorded in DMSO- $d_6$  using tetramethylsilane as an internal reference on BRUKER AVANCE II 400 NMR spectrometer from CIL, Guru Jambheshwar University of Science and Technology, Hissar, Haryana, India.

#### Preparation of OrganylTellurium(IV) Trichlorides and DiorganylTellurium(IV) Dichlorides

4-Methoxyphenyltellurium(IV) trichloride[62,63], bis(p-methoxyphenyl)tellurium(IV) dichloride[63,64], 4-hydroxyphenyltellurium(IV) trichloride[65], bis(p-hydroxyphenyl)tellurium(IV) dichloride[65], 3-methyl-4-hydroxyphenyltellurium(IV) trichloride[66] and bis(3-methyl-4-hydroxyphenyl)tellurium(IV) dichloride[66] were prepared by the reactions of tellurium tetrachloride (Aldrich) with corresponding arenes i.e. anisole, phenol, o-cresol respectively, by the methods reported in the literature[62-66].

#### Preparation of 5-Chlorosalicylaldehyde-3-Aminopyridine Schiff base (3-APY-{5-ClSalH})

The Schiff base was prepared by mixing equimolecular quantity of 5-chlorosalicylaldehyde (0.08 mole, 12.53g) and 3-aminopyridine (0.08 mole, 7.52g) in 25 ml methanol in a round bottomed flask equipped with a condenser[67]. The reaction mixture was refluxed on water bath for 4 hours. After completion of reaction, the reaction mixture was cooled, filtered and dried in a desiccator over anhydrous  $CaCl_2$  and recrystallized from methanol, a sharp orange crystalline product was obtained.

#### Preparation of Complexes

Tellurium tetrachloride, organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides, when reacted with Schiff base form solid complexes as described below:

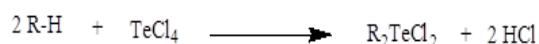
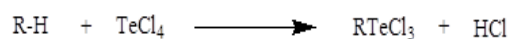
$[TeCl_3(3-APY-\{5-ClSal\})]$ ,  $[R_2TeCl_2(3-APY-\{5-ClSal\})]$  and  $[R_2TeCl(3-APY-\{5-ClSal\})]$

The solid complexes were prepared by addition of 5 mmol tellurium(IV) derivatives in about 25 mL

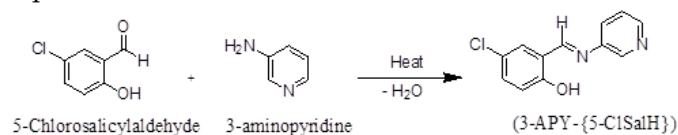
anhydrous methanol to a hot solution of 5 mmol Schiff base (3-APY-{5-ClSalH}) in about 25 mL methanol with continuous stirring. The reaction mixture was refluxed on steam bath for 4 hours. The excess solvent was distilled off to obtain the desired products which were recrystallized from dry methanol. The coloured complexes crystallized out, which were filtered, washed with dry methanol and dried in a vacuum desiccator over  $P_4O_{10}$ .

### III. RESULTS AND DISCUSSION

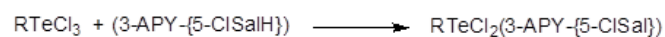
$TeCl_4$  when heated with anisole[62-64], phenol[65] and o-cresol[66] (R-H) appears to undergo the Friedel Craft type condensation reaction where by  $TeCl_4^+$  unit attacks a position para to the methoxy / hydroxyl group in the aromatic ring, thus resulting in the formation of organyltellurium(IV) trichlorides and diorganyltellurium(IV) dichlorides.



Preparation of Schiff Base (3-APY-{5-ClSalH}), by the reaction of 5-chlorosalicylaldehyde with 3-aminopyridine can be represented by following equation.



Schiff Base reacts with tellurium (IV) chloride, organyltellurium (IV) trichlorides and diorganyltellurium (IV) dichlorides to yield the coloured tellurium (IV) complexes.



All the tellurium (IV) complexes are colored, crystalline solids, stable at room temperature and non-hygroscopic in nature. They are insoluble in non

polar and less polar organic solvents, but are soluble in polar donor solvents like DMF, DMSO etc. The analytical data along with their physical properties are presented in Table 1.

### Conductance Studies

Molar conductance ( $\Lambda_M$ ) data for organyltellurium(IV) Schiff base complexes in DMSO are compiled in Table 1. The  $\Lambda_M$  values at ca.  $10^{-3}$  M of complexes lies in the range 8.97-48.97 S  $\text{cm}^2 \text{mol}^{-1}$  which predict the non-electrolyte to 1:1 weak electrolyte type behavior[68,69] of these Schiff base complexes in DMSO, probably due to ionization into  $\text{TeCl}_2(3\text{-APY-}\{5\text{-ClSal}\})^+ / \text{RTeCl}(3\text{-APY-}\{5\text{-ClSal}\})^+ / \text{R}_2\text{Te}(3\text{-APY-}\{5\text{-ClSal}\})^+$  and  $\text{Cl}^-$  in DMSO. The higher  $\Lambda_M$  values for some complexes may be due to steric factors and donor behavior of DMSO to result in probable dissociation into solvated cation and 3-APY- $\{5\text{-ClSal}\}$  along with  $\text{Cl}^-$  in DMSO. This conductance behavior of tellurium(IV) Schiff base complexes is different from those of transition metal complexes[70] which are reported to be non-electrolytes.

### Infrared Spectra

In order to give conclusion idea about the structure of metal complexes, the main IR bands were compared with those of free ligands (Table 2).

In the IR spectra of the ligand show the sharp bands[31,38,41,67,71], one at  $1626 \text{ cm}^{-1}$  of azomethine  $\nu(\text{C}=\text{N})$  group and another at  $3070 \text{ cm}^{-1}$ , of phenolic  $\nu(\text{OH})$  group. In all the complexes  $\nu(\text{C}=\text{N})$  bands were shifted to downward region[22,38,41,67,71-73] i.e.  $1604\text{-}1615 \text{ cm}^{-1}$  confirms the tellurium coordination through azomethine nitrogen[74,75]. The absence of peak due to the phenolic  $-\text{OH}$  group in free ligand at  $3070 \text{ cm}^{-1}$  in the complexes suggests the coordination of ligand to the metal through phenolic oxygen via deprotonation which infers that azomethine nitrogen and phenolic oxygen are the coordination sites of bidentate ligand.

In all the tellurium complexes additional bands are also appear in the region at  $280\text{-}293 \text{ cm}^{-1}$ [38,41,72,73,76,77] and in the range of  $414\text{-}419 \text{ cm}^{-1}$ [78]. These bands have been ascribed to  $\nu(\text{Te-O})$  and  $\nu(\text{Te-N})$  respectively, indicating coordination through oxygen and nitrogen atom of Schiff base.

Further, presence of organyl groups of RTe and  $\text{R}_2\text{Te}$  may result in mixing of certain bands, thus making independent assignments very difficult. Thus, the Schiff base ligand is coordinated to the tellurium atom as uninegative ON bidentate ligand give rise to a six membered chelating ring with tellurium centre.

**Table 1.** Analytical Data, Molar Conductance and Physical Properties for Schiff Base (3-APY- $\{5\text{-ClSalH}\}$ ) Complexes of Tellurium(IV)

Compound No.	Complex (R)	Empirical Formula (Formula Wt.)	Colour (Yield,%)	M. Pt. ( $^{\circ}\text{C}$ ) dec.	Analyses % Found (Calculated)					$\Lambda_M$ at ca. $10^{-3}\text{M}$ S $\text{cm}^2\text{mol}^{-1}$ in DMSO
					C	H	N	Te	Cl	
Schiff Base	(3-APY- $\{5\text{-ClSalH}\}$ )	$\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}$ (232.64)	Orange (86)	80-82	61.67 (61.95)	3.72 (3.87)	11.95 (12.04)	-	15.04 (15.26)	-
1	$\text{TeCl}_2(3\text{-APY-}\{5\text{-ClSal}\})$	$\text{C}_{12}\text{H}_8\text{Cl}_4\text{N}_2\text{O}_2\text{Te}$ (465.74)	Dark Yellow (81)	158-160	30.65 (30.94)	1.65 (1.72)	5.91 (6.02)	27.12 (27.40)	30.23 (30.49)	8.97
2	$\text{RTeCl}_2(3\text{-APY-}\{5\text{-ClSal}\})$ (4-methoxyphenyl)	$\text{C}_{19}\text{H}_{15}\text{Cl}_3\text{N}_2\text{O}_2\text{Te}$ (537.31)	Cream (73)	136-138	42.26 (42.47)	2.53 (2.79)	5.04 (5.21)	23.89 (23.75)	19.67 (19.82)	24.69
3	$\text{RTeCl}_2(3\text{-APY-}\{5\text{-ClSal}\})$ (4-hydroxyphenyl)	$\text{C}_{18}\text{H}_{13}\text{Cl}_3\text{N}_2\text{O}_2\text{Te}$ (523.30)	Dark Yellow (79)	216-218	41.07 (41.31)	2.25 (2.48)	5.22 (5.35)	24.56 (24.38)	20.12 (20.35)	29.03
4	$\text{RTeCl}_2(3\text{-APY-}\{5\text{-ClSal}\})$ (3-methyl-4-hydroxyphenyl)	$\text{C}_{19}\text{H}_{15}\text{Cl}_3\text{N}_2\text{O}_2\text{Te}$ (537.31)	Light Grey (72)	196-198	42.21 (42.47)	2.65 (2.79)	5.08 (5.21)	23.57 (23.75)	19.67 (19.82)	19.11
5	$\text{R}_2\text{TeCl}(3\text{-APY-}\{5\text{-ClSal}\})$ (4-methoxyphenyl)	$\text{C}_{26}\text{H}_{22}\text{Cl}_2\text{N}_2\text{O}_2\text{Te}$ (608.88)	Light Orange (80)	232-234	51.03 (51.28)	3.45 (3.61)	4.44 (4.60)	29.82 (20.96)	11.48 (11.66)	32.55
6	$\text{R}_2\text{TeCl}(3\text{-APY-}\{5\text{-ClSal}\})$ (4-hydroxyphenyl)	$\text{C}_{24}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_2\text{Te}$ (580.86)	Light Brown (84)	209-211	49.46 (49.62)	2.97 (3.10)	4.58 (4.82)	21.78 (21.97)	12.03 (12.22)	44.52
7	$\text{R}_2\text{TeCl}(3\text{-APY-}\{5\text{-ClSal}\})$ (3-methyl-4-hydroxyphenyl)	$\text{C}_{26}\text{H}_{22}\text{Cl}_2\text{N}_2\text{O}_2\text{Te}$ (608.88)	Light Yellow (86)	222-224	51.06 (51.28)	3.47 (3.61)	4.43 (4.60)	20.74 (20.96)	11.42 (11.66)	48.97

Values of  $\Lambda_M$  reported<sup>68, 69</sup> for 1:1 electrolytes in DMSO =  $50 - 70 \text{ S cm}^2 \text{mol}^{-1}$

**Table 2.** Important IR Data (cm<sup>-1</sup>) of the Schiff Base (3-APY-{5-ClSalH}) and complexes

(s = strong, m = medium, b = broad, sh = shoulder)

*<sup>1</sup>H NMR Spectra*

Proton NMR Spectral data of Schiff base and all its complexes are compiled in Table 3.

Schiff base exhibits signals at 12.754, 8.587 and 6.989-7.589 δ ppm which are assigned to phenolic -OH, azomethine proton -N=CH- and aromatic as well as pyridine protons respectively.

The proton peak of the -OH group had disappeared, which suggests that the hydroxyl group coordinates to tellurium centre. Also the signal at 8.587 δ ppm attributed to imine hydrogen in free ligand, which is downfield shifting in the complexes, which proves that imine nitrogen took part in the coordination[38,41,51,52,71,79].

Independent assignments to the aryl protons of (3-APY-{5-ClSalH}) and RTe / R<sub>2</sub>Te are not possible due to overlapping of signals in this region.

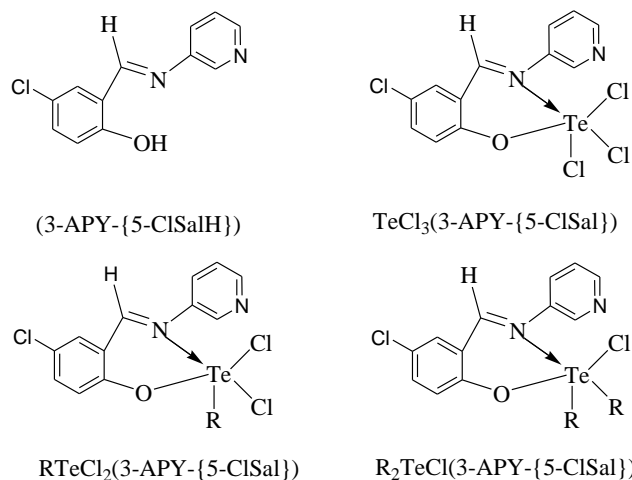
Compound No.	(Phenolic) $\nu_{(OH)}$	(Azomethine) $\nu_{(C=N)}$	$\nu_{(Te-O)}$	$\nu_{(Te-N)}$
(3-APY-{5-ClSalH})	3070 b	1626 s	-	-
1	-	1604 sh	290 s	419 s
2	-	1609 s	290 s	419 s
3	-	1615 s	289 s	419 s
4	-	1607 s	280 s	418 s
5	-	1608 s	289 s	419 s
6	-	1615 s	289 s	419 s
7	-	1615 s	293 s	414 b

**Table 3.** <sup>1</sup>H NMR Spectral Data of Schiff Base (3-APY-{5-ClSalH}) and Complexes in DMSO-d<sub>6</sub>.

Compound Number	(Phenolic) -OH $\delta$ ppm	(Azomethine) -HC=N $\delta$ ppm	(Ar rings protons) $\delta$ ppm	-CH <sub>3</sub> /-OCH <sub>3</sub> $\delta$ ppm	-OH of RTe/R <sub>2</sub> Te $\delta$ ppm
(3-APY-{5-ClSalH})	12.754 (s, 1H)	8.587 (s, 1H)	6.989-7.589 (cm, 7H)	-	-
1	-	10.269 (s, 1H)	6.915-8.019 (cm, 7H)	-	-
2	-	10.254 (s, 1H)	6.954-8.012 (cm, 11H)	4.181 (s,3H)	-
3	-	10.272 (s, 1H)	6.875-8.100 (cm, 11H)	-	9.110 (s,1H)
5	-	10.270 (s, 1H)	6.910-8.036 (cm, 15H)	3.820 (s,6H)	-
6	-	10.271 (s, 1H)	6.840-8.235 (cm, 15H)	-	9.109 (s,2H)
7	-	10.139 (s, 1H)	7.717-8.150 (cm, 13H)	2.508 (s,6H)	9.067 (s,2H)

s = singlet, cm = complex multipl. Spectra of compound number 4 not well resolved due to poor solubility

On the basis of spectral studies, resulting in the formation of pentacoordinated tellurium(IV) complexes and proposed structures are shown in Figure 1.



**Figure 1:** Proposed Structures of Schiff base (3-APY-{5-ClSalH}) and Tellurium (IV) Complexes.

### Biological studies

Schiff base and their tellurium(IV) complexes were screened[80] against two Gram-positive bacteria (*S. aureus* MTCC 96 and *S. pyogenes* MTCC 442), two Gram-negative bacteria (*P. aeruginosa* MTCC 1688 and *E. coli* MTCC 443) and antifungal activity against three fungal strains *C. albicans* MTCC 227, *A. niger* MTCC 282 and *A. clavatus* MTCC 1323.

The factors that govern antibacterial activities are strongly dependent on the central metal ion and the coordination numbers and also due to the presence of nitrogen and sulfur donor groups[81-83].

**Table 4.** Minimum Inhibitory Concentration MIC ( $\mu\text{g/mL}$ ) of Schiff Base (3-APY-{5-ClSalH}) and Complexes

Compound Number	Bacterial Strain				Fungal Strain		
	<i>S. aureus</i> MTCC 96	<i>S. pyogenes</i> MTCC 442	<i>P. aeruginosa</i> MTCC 1688	<i>E. coli</i> MTCC 443	<i>C. albicans</i> MTCC 227	<i>A. niger</i> MTCC 282	<i>A. clavatus</i> MTCC 1323
(3-APY-{5-ClSalH})	200	200	250	200	500	1000	1000
1	500	500	100	200	1000	> 1000	> 1000
2	250	200	125	100	250	> 1000	> 1000
3	250	125	200	250	250	500	1000
6	100	125	250	250	500	1000	1000
Standard Drugs							
<i>Ampicillin</i>	250	100	100	100	-	-	-
<i>Chloramphenicol</i>	50	50	50	50	-	-	-
<i>Nystatin</i>	-	-	-	-	100	100	100
<i>Greseofulvin</i>	-	-	-	-	500	100	100

In comparison to Schiff base, the tellurium(IV) complexes shows higher biological activities. The complexes i.e 1, 2 and 3 i.e.  $\text{TeCl}_3(3\text{-APY}\{-5\text{-ClSal}\})$ ,  $\text{RTeCl}_2(3\text{-APY}\{-5\text{-ClSal}\})$  and  $\text{RTeCl}_2(3\text{-APY}\{-5\text{-ClSal}\})$ , whereas R = 4-methoxyphenyl, 4-hydroxyphenyl respectively, show substantial activity against bacterial strains (*P. aeruginosa*). The data is given in Table 4.

### IV. CONCLUSION

The Schiff base (3-APY-{5-ClSalH}) has been prepared by condensation of 5-chlorosalicylaldehyde with 3-aminopyridine. Schiff base when reacted with tellurium tetrachloride, organytellurium(IV) trichlorides and diorganytellurium(IV) dichlorides in 1:1 molar ratios yield  $[\text{TeCl}_3(3\text{-APY}\{-5\text{-ClSal}\})]$ ,  $[\text{RTeCl}_2(3\text{-APY}\{-5\text{-ClSal}\})]$  and  $[\text{R}_2\text{TeCl}(3\text{-APY}\{-5\text{-ClSal}\})]$ : where R = 4-methoxyphenyl, 4-

hydroxyphenyl, 3-methyl-4-hydroxyphenyl type complexes. Spectral studies predict the pentacoordinated tellurium centre by the monobasic bidentate (ON) Schiff base. Some of these complexes possess substantial antimicrobial activity.

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